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FIG. 1A

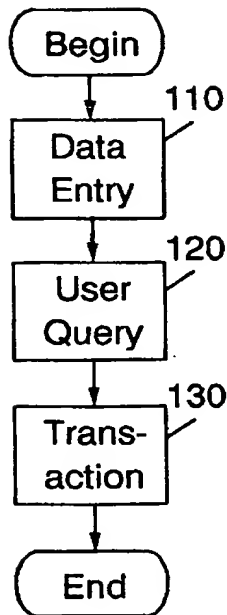


FIG. 1B

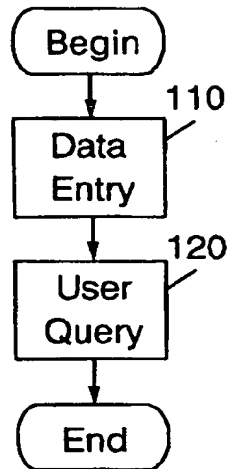


FIG. 1C

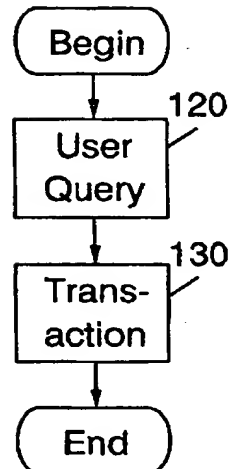


FIG. 1D

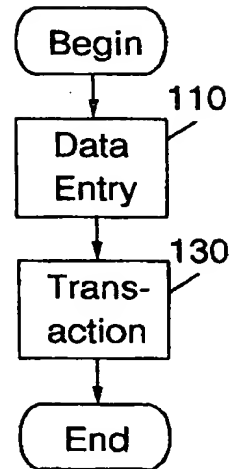


FIG. 1E

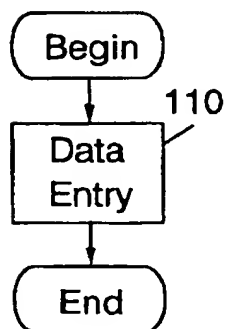


FIG. 1F

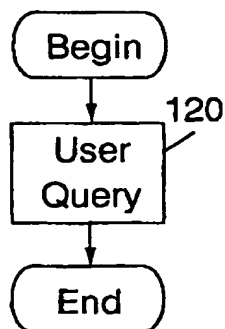


FIG. 1G

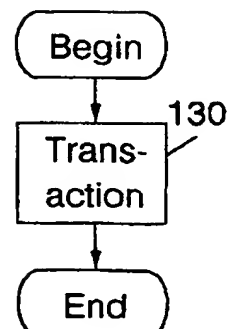


FIG. 2

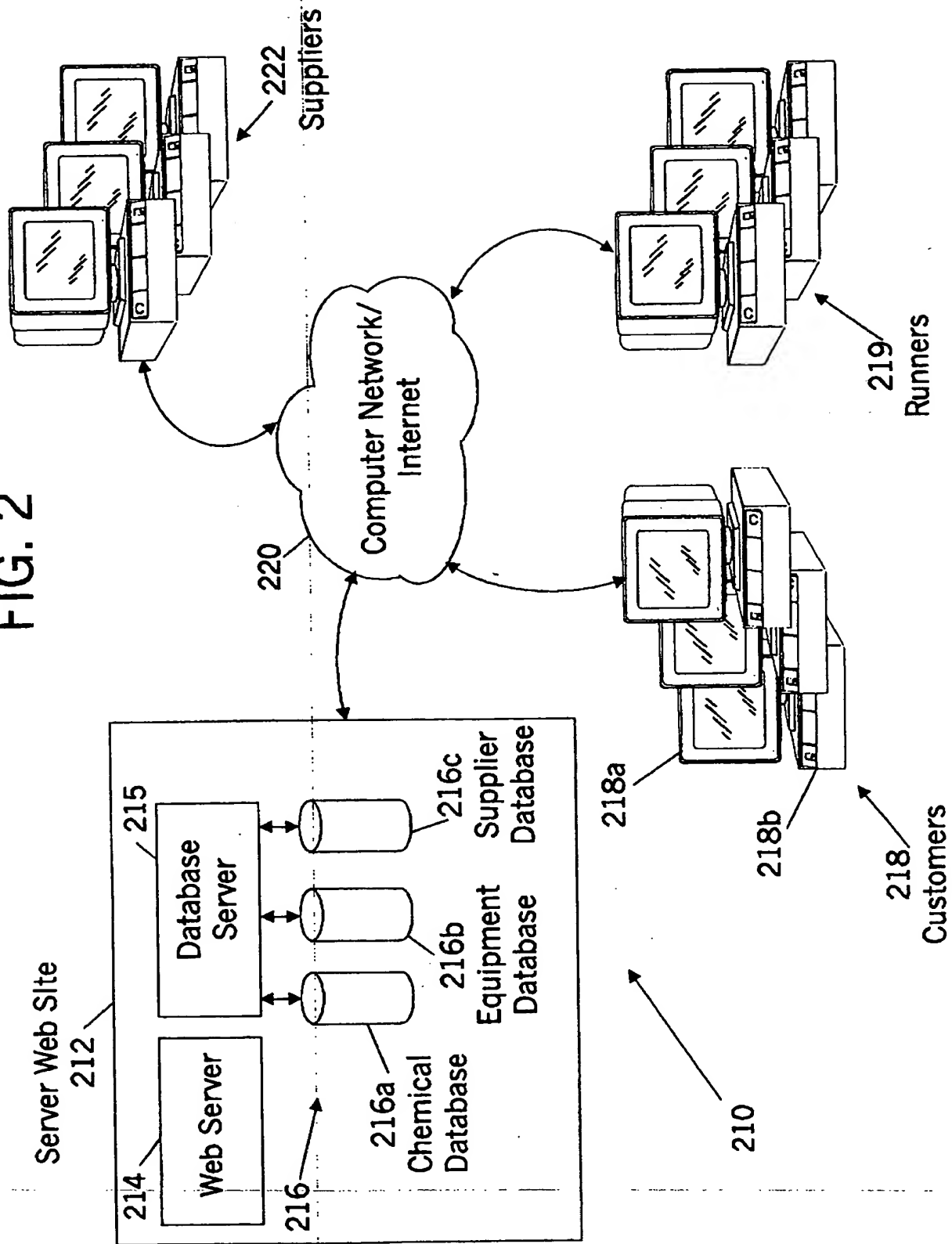
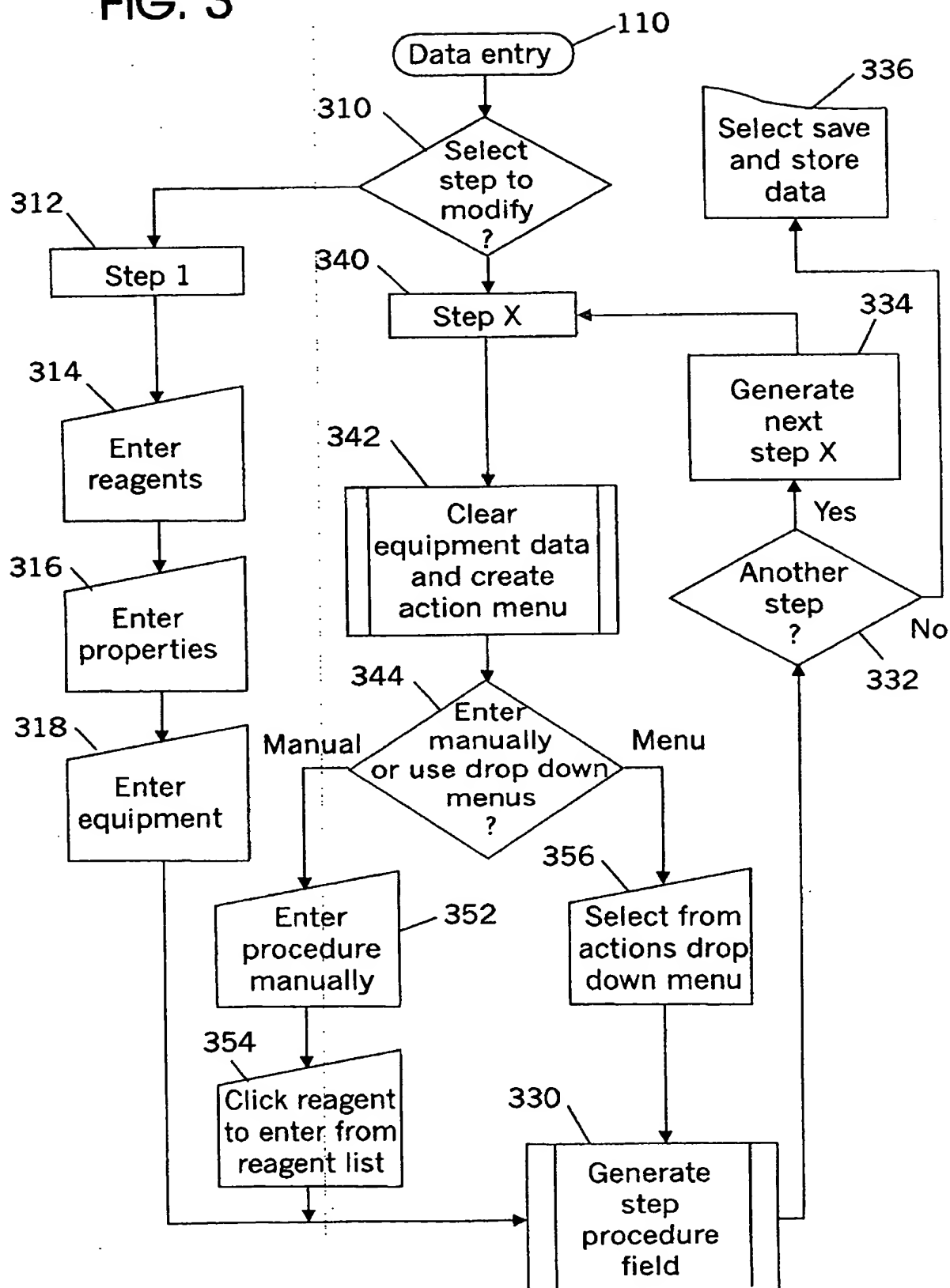


FIG. 3



Synthemalix Protocol Manager ver. 0.90b

Name:  CAS:  Formula:  Weight:

Reagents:

|    | CAS                  | Name                 | Weight               | Amount (g)           |
|----|----------------------|----------------------|----------------------|----------------------|
| 1  | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> |
| 2  | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> |
| 3  | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> |
| 4  | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> |
| 5  | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> |
| 6  | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> |
| 7  | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> |
| 8  | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> |
| 9  | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> |
| 10 | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> |

Starting Flask:

Equipped with:

- 
- 
- 
- 
- 
- 
- 
- 
- 
- 

Procedure

Step:

Reaction Type/Keywords (Separated by semicolon)

FIG. 4

Synthematix Protocol Manager ver. 0.90b

Name: \_\_\_\_\_ CAS: \_\_\_\_\_ Formula: \_\_\_\_\_ Weight: \_\_\_\_\_

Reagents:   ☐ Enter procedure by hand

**Search Results**

| CAS        | Chemical Name                    |
|------------|----------------------------------|
| 71-43-2    | Benzene                          |
| 104-86-9   | Benzenemethanamine, 4-chloro-    |
| 105-13-5   | Benzenemethanol, 4-methoxy-      |
| 105-05-5   | Benzene, 1,4-diethyl-            |
| 118-69-4   | Benzene, 1,3-dichloro-2-methyl-  |
| 121-14-2   | Benzene, 1-methyl-2,4-dinitro-   |
| 70-34-8    | Benzene, 1-fluoro-2,4-dinitro-   |
| 93-05-0    | 1,4-Benzenediamine, N,N-diethyl- |
| 98-10-2    | Benzenesulfonamide               |
| 104-51-8   | Benzene, butyl-                  |
| 827-52-1   | Benzene, cyclohexyl-             |
| 14321-27-8 | Benzenemethanamine, N-ethyl-     |

**Procedure**

Step 2

|     |                      |     |                      |
|-----|----------------------|-----|----------------------|
| Qty | <input type="text"/> | Qty | <input type="text"/> |
| Qty | <input type="text"/> | Qty | <input type="text"/> |
| Qty | <input type="text"/> | Qty | <input type="text"/> |
| Qty | <input type="text"/> | Qty | <input type="text"/> |
| Qty | <input type="text"/> | Qty | <input type="text"/> |

FIG. 5

FIG. 5

**Synthematix Protocol Manager ver. 0.90b**

Name:

**Reagents**

| CAS      | Name | Weight | Formula | Density | BP | FP | MP | Vapor Pressure | Comments | Beilstein | Other Names |
|----------|------|--------|---------|---------|----|----|----|----------------|----------|-----------|-------------|
| 101-01-1 |      |        |         |         |    |    |    |                |          |           |             |
|          |      |        |         |         |    |    |    |                |          |           |             |
|          |      |        |         |         |    |    |    |                |          |           |             |
|          |      |        |         |         |    |    |    |                |          |           |             |
|          |      |        |         |         |    |    |    |                |          |           |             |
|          |      |        |         |         |    |    |    |                |          |           |             |
|          |      |        |         |         |    |    |    |                |          |           |             |
|          |      |        |         |         |    |    |    |                |          |           |             |
|          |      |        |         |         |    |    |    |                |          |           |             |
|          |      |        |         |         |    |    |    |                |          |           |             |

**Procedure**

Previous  Next

New  Save  Update

Save  Cancel

Qty   Exit

FIG. 6



Synthematix Protocol Manager ver. 0.90b

Name:  CAS:  Formula:  Weight:

Reagents:   ☐ Enter procedure by hand

|    | CAS                  | Name                 | Weight               | Amount (g)           | Action               |
|----|----------------------|----------------------|----------------------|----------------------|----------------------|
| 1  | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> |
| 2  | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> |
| 3  | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> |
| 4  | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> |
| 5  | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> |
| 6  | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> |
| 7  | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> |
| 8  | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> |
| 9  | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> |
| 10 | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> |

Qualifier:

Action:

Reagent:

Time:

Procedure

Step 2

Yield:  Density:

BP:  MP:

MP:  Vapor Pr:

Ballistien:  Other Names:

FIG. 7



Synthematrix Protocol Manager ver. 0.90b

Name: \_\_\_\_\_ CAS: \_\_\_\_\_ Formula: \_\_\_\_\_ Weight: \_\_\_\_\_

Reagents:   ☐ Enter procedure by hand

|    | CAS | Name | Weight | Amount (g) | Action |
|----|-----|------|--------|------------|--------|
| 1  |     |      |        |            |        |
| 2  |     |      |        |            |        |
| 3  |     |      |        |            |        |
| 4  |     |      |        |            |        |
| 5  |     |      |        |            |        |
| 6  |     |      |        |            |        |
| 7  |     |      |        |            |        |
| 8  |     |      |        |            |        |
| 9  |     |      |        |            |        |
| 10 |     |      |        |            |        |

Qualifier:  Action:

Reagent:  Time:

Procedure

Step 2

|     |                      |     |                      |
|-----|----------------------|-----|----------------------|
| Qty | <input type="text"/> | Qty | <input type="text"/> |
| Qty | <input type="text"/> | Qty | <input type="text"/> |
| Qty | <input type="text"/> | Qty | <input type="text"/> |
| Qty | <input type="text"/> | Qty | <input type="text"/> |
| Qty | <input type="text"/> | Qty | <input type="text"/> |

FIG. 8

106340 6326/50

Synthemalix Protocol Manager ver. 0.90b

Name: CAS Formula Weight

Reagents:   ☐ Enter procedure by hand

|    | CAS | Name | Weight | Amount (g) | Action |
|----|-----|------|--------|------------|--------|
| 1  |     |      |        |            |        |
| 2  |     |      |        |            |        |
| 3  |     |      |        |            |        |
| 4  |     |      |        |            |        |
| 5  |     |      |        |            |        |
| 6  |     |      |        |            |        |
| 7  |     |      |        |            |        |
| 8  |     |      |        |            |        |
| 9  |     |      |        |            |        |
| 10 |     |      |        |            |        |

**Search Results**

- Class stir rod
- Overhead stirrer
- Teflon stir paddle

**Procedure**

| Qty                  |      | Qty                  |  |
|----------------------|------|----------------------|--|
| <input type="text"/> | stir | <input type="text"/> |  |
| <input type="text"/> |      | <input type="text"/> |  |
| <input type="text"/> |      | <input type="text"/> |  |
| <input type="text"/> |      | <input type="text"/> |  |
| <input type="text"/> |      | <input type="text"/> |  |

FIG. 9

TOP SECRET

Synthetix Protocol Manager ver. 0.90q

File Apparatus Atmosphere Operations

Name: 6-(allylamino)-5-amino-4-chloropyrimidine CAS: R00-02-3 Formula: C7H9N4Cl Weight: 184.63

Reagents: benzene

|    | CAS      | Name                           | Weight | Amount (g) |
|----|----------|--------------------------------|--------|------------|
| 1  | R00-02-4 | 5-amino-4,6-dichloropyrimidine | 163.99 | 50         |
| 2  | 107-11-9 | 2-Propan-1-amine               | 57.09  | 100        |
| 3  | 64-17-5  | Ethanol                        | 46.07  | 100        |
| 4  | 71-43-2  | Benzene                        | 78.11  | 3146.4     |
| 5  |          |                                |        |            |
| 6  |          |                                |        |            |
| 7  |          |                                |        |            |
| 8  |          |                                |        |            |
| 9  |          |                                |        |            |
| 10 |          |                                |        |            |

Starting Flask: Round bottom 3-neck flask ☒

Equipped with:

|  |                          |
|--|--------------------------|
|  | <input type="checkbox"/> |
|  | <input type="checkbox"/> |
|  | <input type="checkbox"/> |
|  | <input type="checkbox"/> |
|  | <input type="checkbox"/> |
|  | <input type="checkbox"/> |
|  | <input type="checkbox"/> |
|  | <input type="checkbox"/> |
|  | <input type="checkbox"/> |
|  | <input type="checkbox"/> |

Procedure

Step 1

Into a round bottom 3-neck flask was added <GN0> grams (<MN0> mol) of 5-amino-4,6-dichloropyrimidine

FIG. 10

20250323 16:50

Synthematix Protocol Manager ver. 0.90q

File Apparatus Atmosphere Operations

Name: 6-(allylamino)-5-amino-4-chloropyrimidine CAS: R00-02-3 Formula: C7H9N4Cl Weight: 184.63

Reagents: benzene  ☐ Enter procedure by hand

|    | CAS      | Name                           | Weight | Amount (g) | Action |
|----|----------|--------------------------------|--------|------------|--------|
| 1  | R00-02-4 | 5-amino-4,6-dichloropyrimidine | 163.99 | 50         |        |
| 2  | 107-11-9 | 2-Propen-1-amine               | 57.09  | 100        |        |
| 3  | 64-17-5  | Ethanol                        | 46.07  | 100        |        |
| 4  | 71-43-2  | Benzene                        | 78.11  | 3146.4     |        |
| 5  |          |                                |        |            |        |
| 6  |          |                                |        |            |        |
| 7  |          |                                |        |            |        |
| 8  |          |                                |        |            |        |
| 9  |          |                                |        |            |        |
| 10 |          |                                |        |            |        |

Qualifier:

Action:

Time:

Procedure

Step: 7

FIG. 11



Synthematix Protocol Manager ver. 0.90q

File Apparatus Atmosphere Operations

Name: 6-(allylamino)-5-amino-4-chloropyrimidine CAS: R00-02-3 Formula: C7H9N4Cl Weight: 184.63

Reagents: benzene  ☐ Enter procedure by hand

| CAS      | Name                           | Weight | Amount (g) |
|----------|--------------------------------|--------|------------|
| R00-02-4 | 5-amino-4,6-dichloropyrimidine | 163.99 | 50         |
| 107-11-9 | 2-Propen-1-amine               | 57.09  | 100        |
| 64-17-5  | Ethanol                        | 46.07  | 100        |
| 71-43-2  | Benzene                        | 78.11  | 3146.4     |
|          |                                |        |            |
|          |                                |        |            |
|          |                                |        |            |
|          |                                |        |            |
|          |                                |        |            |
|          |                                |        |            |

Action:

Qualifier:

Action:

Reagent:

Time:

Procedure

Step 2

To the flask was added <GN1> grams (<MN1> mol) of 2-Propen-1-amine and <GN2> grams (<MN2> mol) of Ethanol

FIG. 12

**Synthematix Protocol Manager ver. 0.90n**

File Apparatus Atmosphere Operations

Name: 6-(allylamino)-5-amino-4-chloropyrimidine CAS: R00-02-3 Formula: C7H9N4Cl Weight: 184.63

Reagents: benzene [Lookup] ☐ Enter procedure by hand

|    | CAS      | Name                           | Weight | Amount (g) |                            |
|----|----------|--------------------------------|--------|------------|----------------------------|
| 1  | R00-02-4 | 5-amino-4,6-dichloropyrimidine | 163.99 | 50         | Action: [ ]                |
| 2  | 107-11-9 | 2-Propen-1-amine               | 57.09  | 100        | Qualifier: [ ]             |
| 3  | 64-17-5  | Ethanol                        | 46.07  | 100        | Action: [ ]                |
| 4  | 71-43-2  | Benzene                        | 78.11  | 3146.4     | Reagent: [ ]               |
| 5  |          |                                |        |            | Time: [ ]                  |
| 6  |          |                                |        |            | [Reset] [Add to Procedure] |
| 7  |          |                                |        |            |                            |
| 8  |          |                                |        |            |                            |
| 9  |          |                                |        |            |                            |
| 10 |          |                                |        |            |                            |

**Procedure**

[Previous] [Next]

Step 2

To the flask was added <GN1> grams (<MN1> mol) of 2-Propen-1-amine and <GN2> grams (<MN2> mol) of Ethanol

[Info] [New] [Properties] [Save] [Reference] [Extra Equip] [Update]

FIG. 13

6333/50

SynthematiX Protocol Manager ver. 0.90b

Name: \_\_\_\_\_ CAS: \_\_\_\_\_ Formula: \_\_\_\_\_ Weight: \_\_\_\_\_

Reagents:   ☐ Enter procedure by hand

|    | CAS | Name | Weight | Amount (g) | Action |
|----|-----|------|--------|------------|--------|
| 1  |     |      |        |            |        |
| 2  |     |      |        |            |        |
| 3  |     |      |        |            |        |
| 4  |     |      |        |            |        |
| 5  |     |      |        |            |        |
| 6  |     |      |        |            |        |
| 7  |     |      |        |            |        |
| 8  |     |      |        |            |        |
| 9  |     |      |        |            |        |
| 10 |     |      |        |            |        |

Qualifier:

Action:

Reagent:

Time:

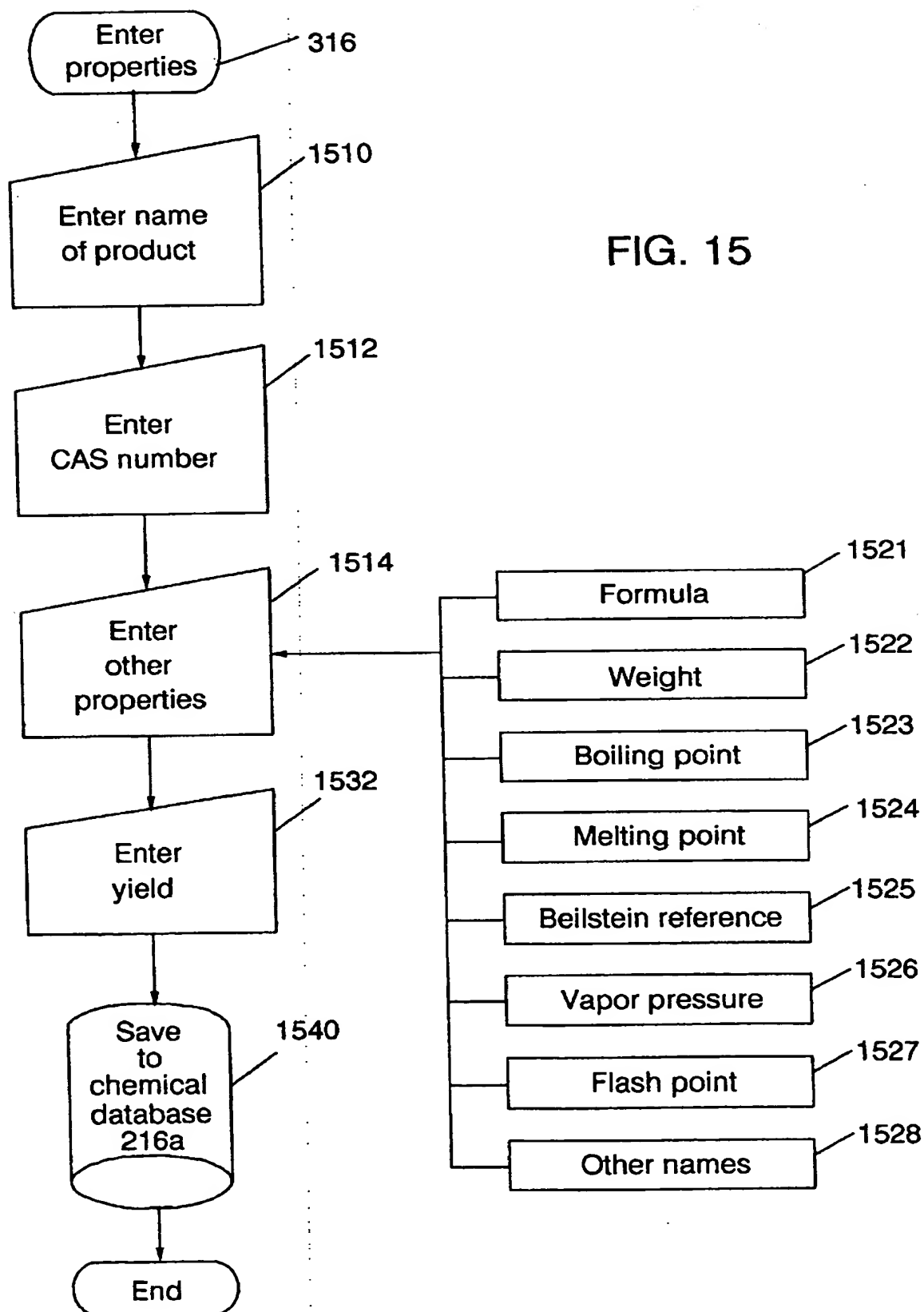
Procedure

Step 2

Reaction Type/Keywords (Separated by semicolon)

FIG. 14





File Apparatus Atmosphere Operations

Name: 6-(ethylamino)-5-amino-4-chloropyrimidine CAS: R00-02-3 Formula: C7H9N4Cl Weight: 184.63

Reagents: benzene  ☐ Enter procedure by hand

| CAS      | Name                           | Weight | Amount (g) | Action                             |
|----------|--------------------------------|--------|------------|------------------------------------|
| R00-02-4 | 5-amino-4,6-dichloropyrimidine | 163.99 | 50         | <input type="button" value="Add"/> |
| 107-11-9 | 2-Propen-1-amine               | 57.09  | 100        | <input type="button" value="Add"/> |

6-(ethylamino)-5-amino-4-chloropyrimidine

Author: Thomas et al. Journal: acid chemistry  
 Year: 1968 Vol: 1  
 Page: 22

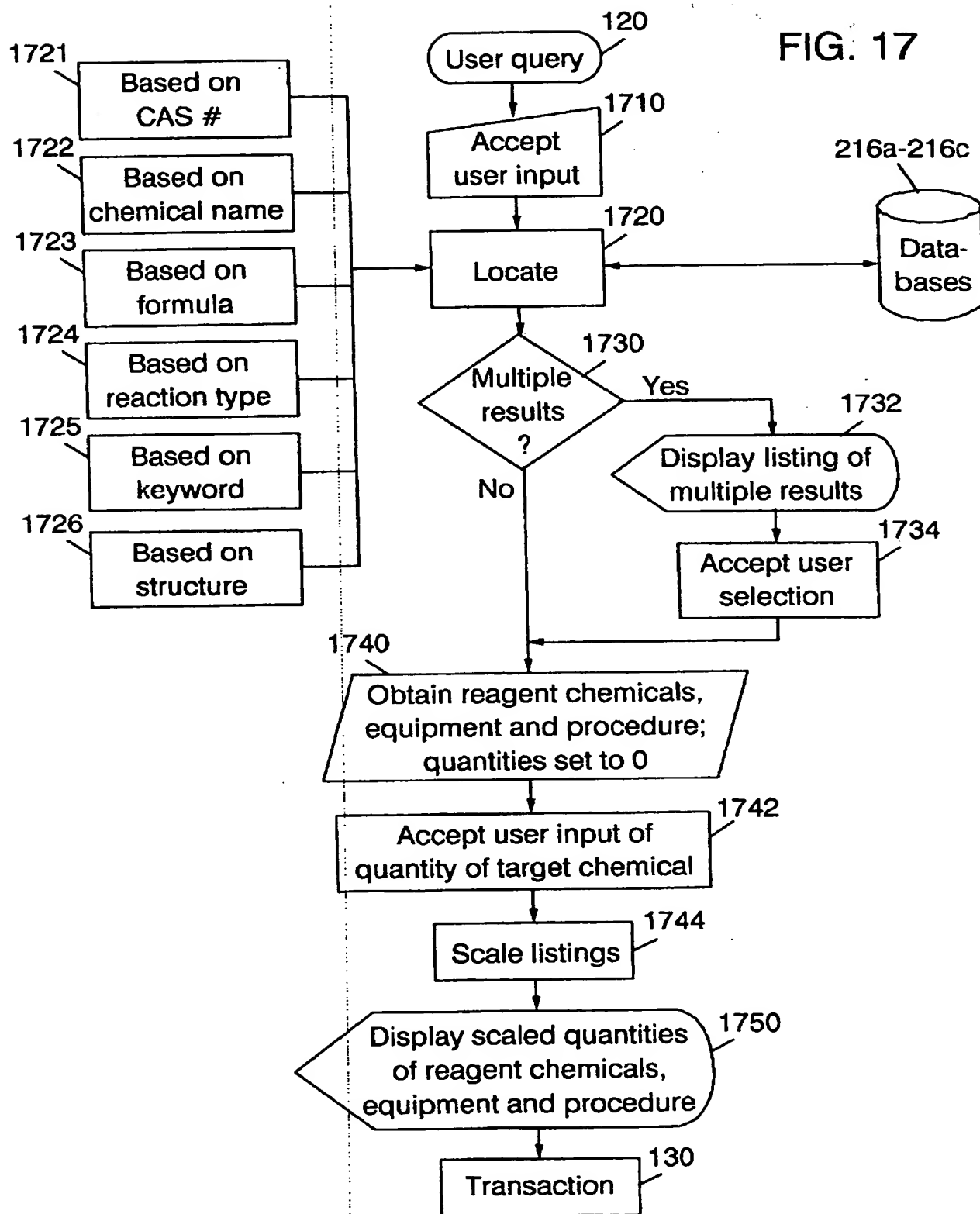
Procedure

Step 6

The extracts are then evaporated to dryness and can be recrystallized from petroleum ether to give a pure product

FIG. 16

FIG. 17




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|---|--|--|

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FIG. 18


Synthematix: Search Results - Netscape

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Bookmarks Location http://63.119.160.203/servlets/QueryProject?name=bromo What's Related

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## Synthematix: Search Results

Number of matches: 7

**alpha-bromo-p-toluene boronic acid**

**n-Butyl-2-bromopropionate**

**(6-Bromo-2,2-diphenylbenzo[1,3]dioxol-5-yl)methanol**

**(6-Bromo-2,2-dimethylbenzo[1,3]dioxol-5-yl)methanol**

**6-bromo-2,2-diphenylbenzo[1,3]dioxole-5-carbaldehyde**

**6-bromo-3,4-[(diphenylmethylene)dioxy]-benzaldehyde ethanediyl acetal**

**1-Benzhydryl-3-bromoazetidine**

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FIG. 19

T06220"6222/60

2010

Synthetic Compound Procedure - Microsoft Internet Explorer

Address: <http://63.119.160.203/servlets/ShowChembase?id=16>

2-Butyl 2-bromopropionate (209.08) C7H13BrO3

2-Bromopropionic Acid  
 $C_4H_7BrO_2$   
 Exact Mass: 151.98  
 Mol. Wt.: 152.98  
 C: 23.55, H: 3.29, Br: 52.23, O: 20.92

1-Butanol  
 $C_4H_{10}O$   
 Exact Mass: 74.07  
 Mol. Wt.: 74.12  
 C: 64.82, H: 13.50, O: 21.59

n-Butyl 2-bromopropionate  
 $C_8H_{17}BrO_2$   
 Exact Mass: 208.01  
 Mol. Wt.: 209.08  
 C: 40.21, H: 6.27, Br: 38.22, O: 15.30

2060

2020

Enter Mol Scale:  mol

Chemicals needed

| Chemical              | Formula | Weight | Equivalent | Moles Needed                        | Grams Needed                        |
|-----------------------|---------|--------|------------|-------------------------------------|-------------------------------------|
| 2-Bromopropionic acid |         | 152.98 | 1          | <input type="text" value="0.0000"/> | <input type="text" value="0.0000"/> |
| n-butanol             |         | 74.12  | 1          | <input type="text" value="0.0000"/> | <input type="text" value="0.0000"/> |
| Hexane                |         | 86.18  | 1          | <input type="text" value="0.0000"/> | <input type="text" value="0.0000"/> |
| Dowex 50W4-200        |         | 3.68   |            | <input type="text" value="0.0000"/> | <input type="text" value="0.0000"/> |
| Water                 |         | 18     | 1          | <input type="text" value="0.0000"/> | <input type="text" value="0.0000"/> |

2030

Equipment needed

| Qty | Equipment                    |
|-----|------------------------------|
| 1   | Round bottom 3-neck flask    |
| 1   | Overhead stirrer             |
| 1   | Dean Stark Trap              |
| 1   | Condenser, Allihn, Drip tip  |
| 1   | Heating mantle               |
| 1   | Recirculating Chiller        |
| 1   | Heavy duty distillation head |
| 1   | Fraction Cutter              |
| 4   | 500 ml round bottom flask    |
| 2   | Varisc                       |
| 3   | Tubing (R.)                  |

2040

Procedure

1. Into a 3 Neck Flask equipped with an overhead stirrer, Dean stark collector, condenser, and heating mantle was placed  grams ( mol) of 2-Bromopropionic acid.
2. To the 2-Bromopropionic acid was added  grams ( mol) of Butanol.
3. To the 2-Bromopropionic acid/butanol mix was added  grams ( mol) of Hexane.
4. To the flask is added  grams of Dowex resin to facilitate the esterification. The resin is previously dried and has an activity of 3.68 grams/mol.
5. Two variscs are attached to the heating mantle and a setting of 50% power is used for heating to rapid reflux.
6. The reaction is monitored for water generation. After collecting  grams ( mol) of water the reaction is complete.
7. Hexane is then removed by continuous draining of the dean stark collector.
8. The reaction is then cooled to room temperature and filtered through a fritted glass funnel to remove the Dowex resin.
9. The crude n-butyl-2-bromopropionate is then purified via vacuum distillation. (The vacuum distillation procedure is below)

Vacuum distillation

- The product is placed into a 5L round bottom and connected to the pilot scale distillation head equipped with a vigorous column. The material is heated in a 5L mantle set to 45-50% power with a varisc.
- The product is collected at a head temp of 40-45°C. The material rapidly condenses once the apparatus is heated up. Total time for distillation is approx. 5 hours.

2050

References

FIG. 20

2010  
2060  
2020  
2030  
2040  
2050

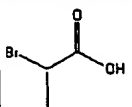
Synthetic Compound Procedure - Microsoft Internet Explorer

Back Forward Stop Refresh Home Search Favorites History Mail Print Edit Discuss Real.com


Address: [?] 7id=16&mol=12&MN0=1835.76&MN1=12&GN1=889.44&MN2=12&GN2=1034.16&MN3=12&GN3=44.16&MN4=12&GN4=216

Links: [?] crn [?] Science resource [?] Internet Sten [?] Windows Update [?] RealPlayer [?] HotBot [?] To Yahoo!

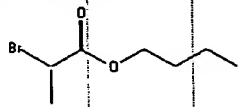
**n-Butyl 2-bromopropionate (209.08)** C7H13BrO3



2-Bromopropionic Acid  
C<sub>3</sub>H<sub>5</sub>BrO<sub>2</sub>  
Exact Mass: 151.95  
Mol Wt: 152.98  
C, 23.55; H, 3.29;  
Br, 52.23; O, 20.92



1-Butanol  
C<sub>4</sub>H<sub>10</sub>O  
Exact Mass: 74.07  
Mol Wt: 74.12  
C, 64.82; H, 13.50; O, 21.59



n-Butyl 2-bromopropionate  
C<sub>7</sub>H<sub>13</sub>BrO<sub>2</sub>  
Exact Mass: 208.01  
Mol Wt: 209.08  
C, 40.21; H, 6.27; Br, 38.22; O, 15.30

+ H<sub>2</sub>O

Enter Mol Scale: [12] mol Calculate

**Chemicals needed**

| Chemical              | Formula Weight | Equivalents | Moles Needed | Grams Needed |
|-----------------------|----------------|-------------|--------------|--------------|
| 2-Bromopropionic acid | 152.98         | 1           | 12.0000      | 1835.7600    |
| n-butanol             | 74.12          | 1           | 12.0000      | 889.4401     |
| Hexane                | 86.18          | 1           | 12.0000      | 1034.1600    |
| Dowex 50W4-200        | 3.68           | 1           | 12.0000      | 44.1600      |
| Water                 | 18             | 1           | 12.0000      | 216.0000     |

**Equipment needed**

| Qty | Equipment                    |
|-----|------------------------------|
| 1   | Round bottom 3-neck flask    |
| 1   | Overhead stirrer             |
| 1   | Dean Stark Trap              |
| 1   | Condenser, Allihn, Drip tip  |
| 1   | Heating mantle               |
| 1   | Recirculating Chiller        |
| 1   | Heavy duty distillation head |
| 1   | Fraction Cutter              |
| 4   | 500 ml round bottom flask    |
| 2   | Varisc                       |
| 3   | Tubing (ft.)                 |

**Procedure**

- Into a 3 Neck Flask equipped with an overhead stirrer, Dean stark collector, condensor, and heating mantle was placed 1835.7600 grams ( 12.0000 mol) of 2-Bromopropionic acid.
- To the 2-Bromopropionic acid was added 889.4401 grams ( 12.0000 mol) of Butanol.
- To the 2-Bromopropionic acid/butanol mix was added 1034.1600 grams ( 12.0000 mol) of Hexane.
- To the flask is added 44.1600 grams of Dowex resin to facilitate the esterification. The resin is previously dried and has an activity of 3.68 grams/mol.
- Two variscs are attached to the heating mantle and a setting of 50% power is used for heating to rapid reflux.
- The reaction is monitored for water generation. After collecting 216.0000 grams ( 12.0000 mol) of water the reaction is complete.
- Hexane is then removed by continuous draining of the dean stark collector.
- The reaction is then cooled to room temperature and filtered through a fritted glass funnel to remove the Dowex resin.
- The crude n-butyl-2-bromopropionate is then purified via vacuum distillation. (The vacuum distillation procedure is below)

**Vacuum distillation**

- The product is placed into a 5L round bottom and connected to the pilot scale distillation head equipped with a vigreux column. The material is heated in a 5L mantle set to 45-50% power with a varisc.
- The product is collected at a head temp of 40-45°C. The material rapidly condenses once the apparatus is heated up. Total time for distillation is approx. 5 hours.

**References**

FIG. 21



FIG. 22

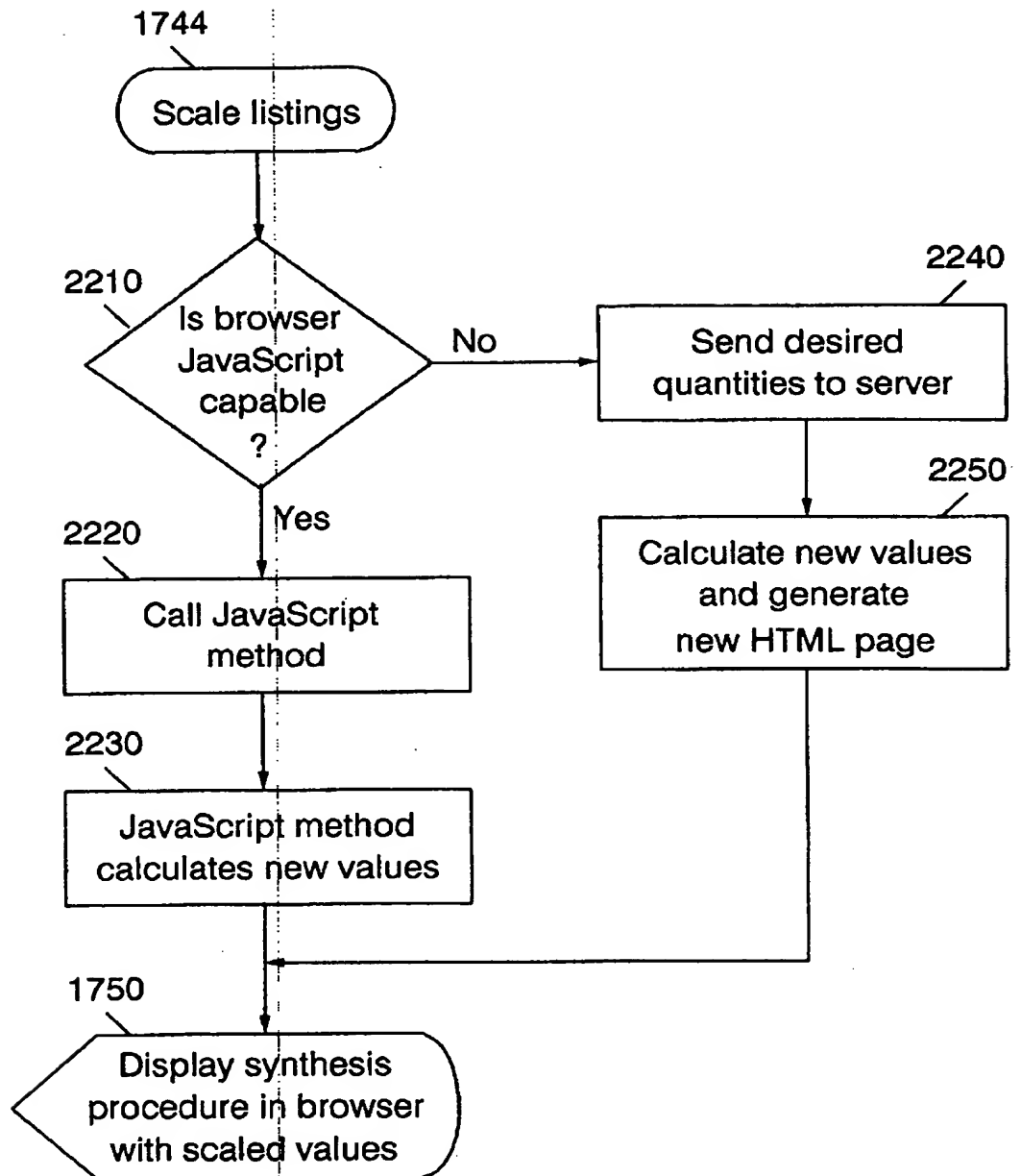


FIG. 23

